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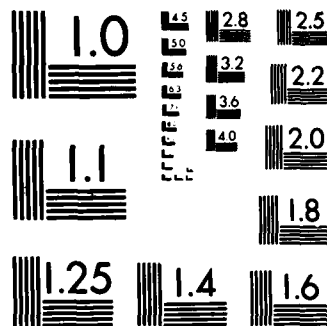
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COLD SINTERING - A NEW POWDER CONSOLIDATION PROCESS

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July 1983

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COLD SINTERING - A NEW POWDER CONSOLIDATION PROCESS

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Abstract

The plastic deformation of powder particles in a high pressure gradient at ambient temperature results in green densities close to theoretical. Physical contact of freshly formed oxide-free surfaces leads to strong particle-particle bonding; this phenomenon is called "cold sintering". High strength levels can be obtained in metals, alloys, composites, ionic and ionic-covalent solids by cold sintering. Subsequent heat treatment of cold sintered material, at temperatures significantly lower than those used in conventional sintering, results in excellent combinations of strength and ductility. Thus, cold sintering offers the potential for retaining metastable constituents, fine-scale microstructures and small precipitates in rapidly solidified powders. Interface reactions or dissolution of phases in metal matrix composites can also be reduced or eliminated. Details of the compacting response, microstructure and mechanical properties of cold sintered and heat treated high speed tool steel and super-alloy powders are presented. Cold sintering characteristics are understood in terms of the interplay of particle shape and particle deformation. Possible cold sintering processing routes of commercial interest are discussed and the limitations of this new technology considered.

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Introduction

High density P/M parts and wrought forms for load-bearing applications are generally fabricated by some form of hot powder consolidation (1). Hot pressing is used in the production of parts of relatively simple shape while hot forging has found acceptance in the powder processing of automotive parts (1,2). Hot pressing, hot extrusion or hot isostatic pressing (HIP) have been utilized in the fabrication of P/M wrought shapes (sheet, bar, tubing...) from beryllium (3), magnesium alloys (4), aluminum alloys (5,6), superalloys (7,8), high speed tool steels (9,10) and dispersion-strengthened alloys (11-13). Typically, these P/M wrought materials exhibit fine-scale microstructures with a homogeneous distribution of precipitates and impurities, resulting in uniformity of mechanical properties.

Rapid solidification processing of powders produces homogeneous fine-scale microstructures and precipitates, highly supersaturated solid solutions, and in the extreme, amorphous metastable structures. Thus, rapid solidification offers technologically important possibilities with respect to the design of new alloys with improved or unique properties. The advantages of rapid solidification processing are reflected in several new aluminum alloy compositions (5,6,14). Typically, rapidly solidified powders with a relatively low oxygen content are produced by some form of inert gas atomization (IGA) or, more recently, by centrifugal atomization (CA), (15).

IGA and CA powders are essentially spherical in shape (1,15,16) and it has been found necessary to resort to hot consolidation at relatively high homologous temperatures to achieve near full density. In contrast, water atomized (WA) powders are more irregular in shape; as a consequence, these powders exhibit higher levels of compressibility and green strength than do spherical powders. When powders are consolidated at relatively high temperatures ($T/T_m \approx 0.6$) there is a high probability that the fine-scale structures and/or metastable constituents developed via rapid solidification will be destroyed. Thus, there is a need to develop methods for the consolidation of alloy powders at the lowest possible temperature, consistent with adequate density and strength.

Cold sintering is one such approach in which plastic deformation of powder particles takes place during compaction at ambient temperature in a gradient of high pressure (17), Figure 1. In this way, densities close to theoretical are achieved with excellent particle to particle bond integrity and associated mechanical properties. Cold sintering (or high pressure consolidation) can be applied to metals and alloys, ionic or ionic-covalent solids; in each, the pressure during compaction exceeds the flow stress of the powder particles. In preliminary studies, cold sintering has been used to consolidate powders of stainless steel (18), refractory metals (19), ferrous alloys (20), aluminum alloys (21), copper and copper-tungsten composites (22) and cadmium telluride (23) at ambient temperatures. It has also been demonstrated that subsequent sintering of the cold sintered compacts at temperatures significantly lower than those used in conventional P/M sintering or hot consolidation, results in high strength and improved ductility.

Consolidation by cold sintering offers unlimited possibilities re the design of new alloys and specialty composite materials which cannot be processed by conventional P/M methods because of decomposition or dissolution of phases at the high consolidation temperatures. Examples include metal-bonded diamond composites (24), steel-carbides, metal-nitrides and metal-oxide composites (17, 20). Since rigid dies are used in cold sintering, and green densities approach 100% theoretical density, close-tolerance parts or shapes can be made. In

conventional P/M processing, this requires coining as an added operation.

The mechanisms of particle consolidation associated with cold sintering have been discussed elsewhere (21,25,26). Transmission electron microscopy of cold sintered pure aluminum powder (25) confirms the importance of plastic deformation of particles in a high pressure gradient. This allows for intimate physical contact of freshly-formed contamination-free surfaces. Fractured oxide layers are dispersed as relatively innocuous small inclusions at particle-particle interfaces. Similar particle behavior is observed in cold sintered elemental copper and iron powders. In terms of green strength, irregularly shaped powders are preferred; for similar powder composition, purity, size and density, cold sintered compacts prepared from irregularly shaped powders exhibit a green strength one order of magnitude higher than cold sintered compacts derived from spherical powders (21,26).

In the present study, a detailed examination has been made of the response of several water atomized irregularly shaped powders to cold sintering, with and without subsequent heat treatment. The materials included were high speed tool steels, and nickel and cobalt-base superalloys. Spherical powders of these alloys would normally require HIP for consolidation to full density. Possible processing routes and limitations using cold sintering are considered, including the production of close-tolerance parts from high performance materials.

Experimental Procedure

Two high speed tool steel compositions were examined, namely T15 and M2. Water atomized (WA) powder of both compositions was obtained from SCM Metal Products (>40%, <44 μ m) and Powdrex Ltd. (~30%, ~44 μ m). Inert gas atomized (IGA) T15 powder made by Crucible Inc. (<6%, ~44 μ m) was also included. All powders were <150 μ m in size. The powders from SCM Metal Products and Powdrex Ltd. had received a vacuum anneal at ~1000°C; oxygen levels were <1000 ppm and <500 ppm for T15 and M2, respectively. The as-atomized (IGA) T15 powder from Crucible Inc. was vacuum annealed at 1000°C prior to cold sintering.

Water atomized Nimonic 80A (a nickel-base superalloy) was supplied by BSA Metal Powders Ltd. and a water-atomized Co-5% Fe alloy by Pfizer, Inc. A limited comparison of cold sintering was made with gas atomized IN-100 and two cobalt-base Stellites supplied by the Cabot Corporation.

With careful design, tool steel dies and punches can be used at pressures up to ~3.0 GPa, i.e. approximately 430,000 psi (26). In the present study, dies and punches were made from T15 P/M tool steel; provided alignment of the tooling is maintained, it is possible to compact powders at pressures up to about 3.5 GPa. To withstand these pressures, the outer diameter of the die is approximately three times larger than the part diameter, Figure 2. Typically, the compact is loaded to ~2 GPa in about 2 seconds; loading is then increased more slowly over a 10-15 second interval to achieve a compaction pressure of >3 GPa. Pressures as high as 4 GPa are possible utilizing cemented carbide dies.

Mechanical properties were assessed by means of hardness and in a three-point bend transverse rupture test. Microstructures and fracture morphologies were characterized utilizing optical and scanning electron microscopy.

Results and Discussion

Compaction

Compaction curves for the two water atomized T15 high speed steel powders are shown in Figure 3. In the case of the IGA spherical T15 IGA powder, compaction response could only be determined at the high end of the pressure scale; at low pressures, the compacts exhibited low green strength. At compaction pressures ≈ 3.0 GPa, the IGA compaction curve was similar to those of the T15 WA powders in Figure 3. It is clear from Figure 3 that densities $>99\%$ of theoretical density can be achieved via cold sintering, if pressures ≈ 3.5 GPa are imposed on the powder. It was found that for a given cold sintered density, the green strength of the compacts prepared from irregularly shaped (WA) powder was significantly higher than that of spherical (IGA) powder.

The cold sintering response of the WA M2 high speed steel powders was similar to that of WA T15. At a comparable cold sintered density, slightly lower compaction pressures were required for M2 compared to T15.

Compaction curves achieved by cold sintering of Nimonic 80A and the Co-5% Fe alloy are given in Figure 4. Green densities $>98\%$ of theoretical density were achieved at compacting pressures ≈ 3.5 GPa. To achieve a density $\approx 90\%$ of theoretical density, the required compacting pressures were ≈ 2.0 GPa and ≈ 2.3 GPa for the Co and Ni-base alloys, respectively. At this level of green density, the pores are essentially isolated. In comparison, spherical IGA powders of nickel-base IN-100, and cobalt-base Stellite #6 and #21 achieved densities $>97\%$ of theoretical density by cold sintering at compacting pressures ≈ 3.5 GPa. Associated green strengths were, however, low for the IGA powders.

Heat Treatment, Structure and Mechanical Properties

To illustrate the heat-treatment response of a cold sintered alloy, the WA T15 high speed tool steel was annealed for 1 hour at 900°C and then heated to 1200°C for 5 minutes and air cooled. Material was then tempered for 1 hour at temperatures up to 800°C . This heat-treatment is similar to the processing and heat-treatment cycle used for commercial P/M T15, i.e. HIP at 1200°C and austenitize at $1225\text{--}1230^\circ\text{C}$ prior to tempering. The tempering curve for the WA T15 cold sintered high speed tool steel is shown in Figure 5. This tempering response is similar to that of IGA HIPed T15, and to that of WA T15 following cold isostatic compaction and vacuum annealing (28). The heat treatment selected in the present study is clearly not an optimum choice; it does, however, demonstrate the viability of using lower heat-treatment temperatures and shorter times to achieve acceptable microstructures.

Cold sintered T15, heat treated as outlined above, and double tempered for 1 hour each time at 550°C exhibited transverse rupture strengths in the range 1800–2000 MPa. This is lower than the level reported for commercial HIP P/M T15, but equivalent to the transverse rupture strength of vacuum sintered T15 processed from WA powder (29) and ingot metallurgy T15. Material cold sintered at 3.5 GPa, annealed for 1 hour at 900°C , repressed at 3.5 GPa and then heat-treated as outlined above achieved transverse rupture strengths in the range 2200–2400 MPa.

A representative fracture surface in the transverse rupture bars is illustrated in Figure 6; the T15 WA powder was cold sintered and heat-treated as

outlined above. Fracture surface morphology reflects both intergranular and ductile fracture modes.

For a given particle size, water atomization produces higher cooling rates than inert gas atomization and hence a finer distribution of carbides. Compared to inert gas atomized T15 powder, the water atomized powder particles are expected to contain a smaller volume fraction of MC carbides (30). The microstructure of a compact cold sintered from a relatively coarse WA T15 powder ($\sim 150\mu\text{m}$) is shown in Figure 7. The fine-scale homogeneous carbide structure is retained after annealing at $\sim 1000^\circ\text{C}$.

The dependence of yield and transverse rupture stress on compacting pressure for Co-5% Fe following a 1 hour anneal at 900°C is shown in Figure 8. There is a significant increase in σ_{TRS} at compacting pressures >2 GPa, corresponding to densities $>95\%$ of theoretical density. Fracture surface morphology in a transverse rupture bar cold sintered at 3.0 GPa and annealed 1 hour at 900°C is shown in Figure 9. This is characteristic of a relatively ductile fracture mode which is a consequence of the sound particle to particle bonding produced by cold sintering and subsequent heat treatment.

The effect of compacting pressure and post cold sintering heat treatment on the transverse rupture strength of WA Nimonic 80A is plotted in Figure 10. Significant increases in σ_{TRS} occur at compacting pressures in the range 2.2-2.5 GPa, at which the density is at least 95% of the theoretical density. σ_{TRS} is a sensitive function of heat treatment; aging produces strength levels ~ 1500 MPa, Figure 10.

A comparison of fracture surfaces of transverse rupture test pieces from Nimonic 80A for post compaction annealing and aging is given in Figures 11(a) and 11(b), respectively. After a 1 hour anneal at 900°C , brittle fracture predominates, with an intergranular propagation mode, Figure 11(a). Double aging after cold sintering results in ductile transgranular fracture, Figure 11(b). The strength of cold sintered ($P = 3$ GPa) and aged Nimonic 80A is higher than that of the aged wrought ingot metallurgy counterpart.

Cold Sintering Process Routes

It has been demonstrated that high densities and excellent mechanical properties may be achieved via cold sintering followed by heat treatment at relatively low temperatures. The impact of cold sintering is most pronounced when irregularly shaped water atomized powders are used. Apart from this advantage in cold sintering, the availability of irregularly shaped particles of low oxygen content should permit the use of lower processing temperatures in conventional hot consolidation. Thus, it is important to explore new methods of inert atmosphere atomization for the production of non-spherical powders.

With respect to rapid solidification technology, cold sintering offers the potential for retaining metastable constituents/phases, and fine scale microstructures and precipitates. It is anticipated that a coupling of rapid solidification processing and cold sintering followed by heat treatment at relatively low temperatures, will allow for the design of new high temperature or wear resistant alloys.

Possible cold sintering processing routes involving rapidly solidified powders with attendant metastable structures are shown schematically in Part 1 of Figure 12. If flow stress decreases markedly with temperature, it may be useful to use "warm" sintering at temperatures somewhat above ambient. Dies and

punches fabricated from high speed tool steel can be used at temperatures up to $\sim 500^{\circ}\text{C}$ with pressures ~ 3 GPa. In this temperature regime, oxidation of the powder is not a major problem. Preliminary observations on the warm sintering of W, Mo and W-Cu composites in the temperature range $200\text{--}300^{\circ}\text{C}$ confirm higher densities and mechanical property levels. Similarly, P/M aluminum alloys are amenable to warm sintering (21,32) at temperatures close to ambient. While the flow stress of Ti5 is essentially constant up to $\sim 500^{\circ}\text{C}$, warm sintering may be advantageous because of the higher ductility. When there is a pronounced decrease in flow stress with temperature, lower cold sintering compacting pressures may be used with an attendant increase in tool life.

The attraction of cold sintering resides in the possibility of designing and producing specialty alloys and composite materials that are unobtainable via conventional P/M or ingot metallurgy - because of decomposition and/or dissolution of constituents at conventional P/M processing temperatures. The approach is shown schematically in Part 2 of Figure 12. A higher volume fraction of carbides, borides, nitrides or oxides may be added to existing hard facing alloy powders to increase abrasive wear resistance. Heat treatment of the cold sintered powder alloys at relatively low temperatures should then result in stable microstructures. Alloying by diffusion can also be envisaged at relatively low heat treatment temperatures with enhanced diffusion via dislocations in heavily deformed material. Resulting microstructures in heavily deformed cold sintered V, Nb and Ta (19) have been shown to be homogeneous with respect to solute atoms and precipitates. In turn, these powder processed refractory metals exhibit high levels of strength and ductility.

For high performance applications, cold sintered parts must have densities close to 100% theoretical density. As discussed previously, the time for loading to maximum compacting pressure is 10-15 seconds. For a complete cold sintering cycle, including die wall lubrication, powder filling, application of pressure, unloading and part ejection, times ~ 1 minute are required. This may be an economically viable rate since cold sintering utilizes specialty materials, resulting in expensive parts. Parallel compaction of several parts will of course increase the rate of production. As an alternative to increasing the production rate, powder preforms can be prepared by conventional compaction technology; the preforms ($\sim 80\%$ theoretical density) are then cold sintered to near full density. Preforms may be cold sintered individually or after stacking in convenient multiples. Part 3 in Figure 12 shows schematically the possible processing route for mass production of high performance parts.

To further enhance mechanical property levels, cold sintered parts or wrought forms can be repressed or HIPed. Since the density after cold sintering is close to theoretical density, HIPing of the cold sintered compacts may be performed at lower temperatures, times and/or pressures. A further advantage is that after cold sintering, voids are in the form of isolated pores so that containers are not required for HIPing.

Limitations of Cold Sintering

The main limitation in cold sintering is part size since large press capacity is mandated. An industrial press of 10,000 ton capacity can be used to generate consolidation pressures ~ 3 GPa in a part up to ~ 200 mm dia. In warm sintering, consolidation pressure will be lower so that larger parts and/or smaller press capacity becomes feasible. Further research is needed in order to determine the efficiency of cold sintering in the production of parts and wrought forms from specialty alloys.

Conclusions

1. Green densities approaching 100% of theoretical density can be achieved by cold sintering in high speed tool steel and superalloy powders of irregular shape.
2. Heat treatment of cold sintered compacts at temperatures significantly lower than those used in conventional P/M processing result in excellent combinations of strength and ductility.
3. Cold sintering offers a means to retain metastable structures in rapidly solidified powders and to fabricate new composite materials. Economically viable production rates for high performance parts appear possible.
4. The major limitation in cold sintering is part size; a large press capacity is mandated since compacting pressures ≈ 3 GPa are required.

Acknowledgment

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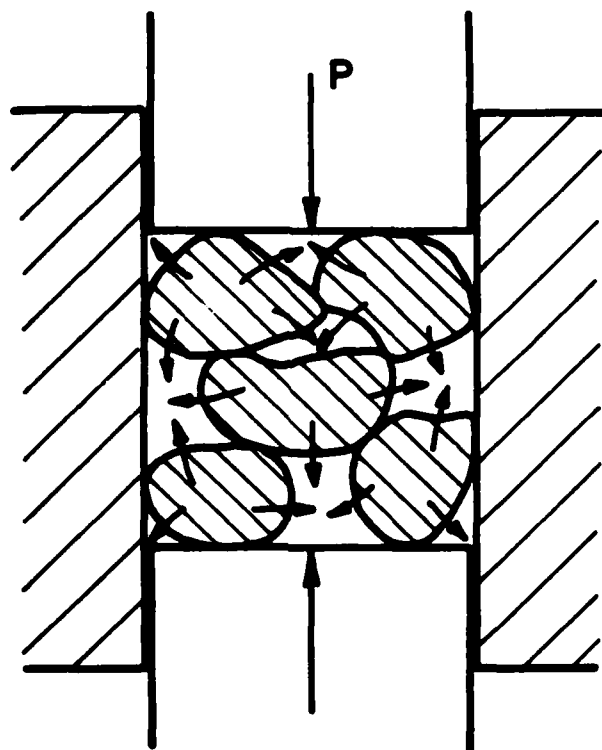


Fig. 1. A schematic drawing of plastic flow of powder during high pressure consolidation.



Fig. 2. Example of a die and punches with cross-section $\approx 20 \text{ cm}^2$ for high pressure consolidation.

Fig. 3. Compaction curves for T15 water atomized high speed steel powder.

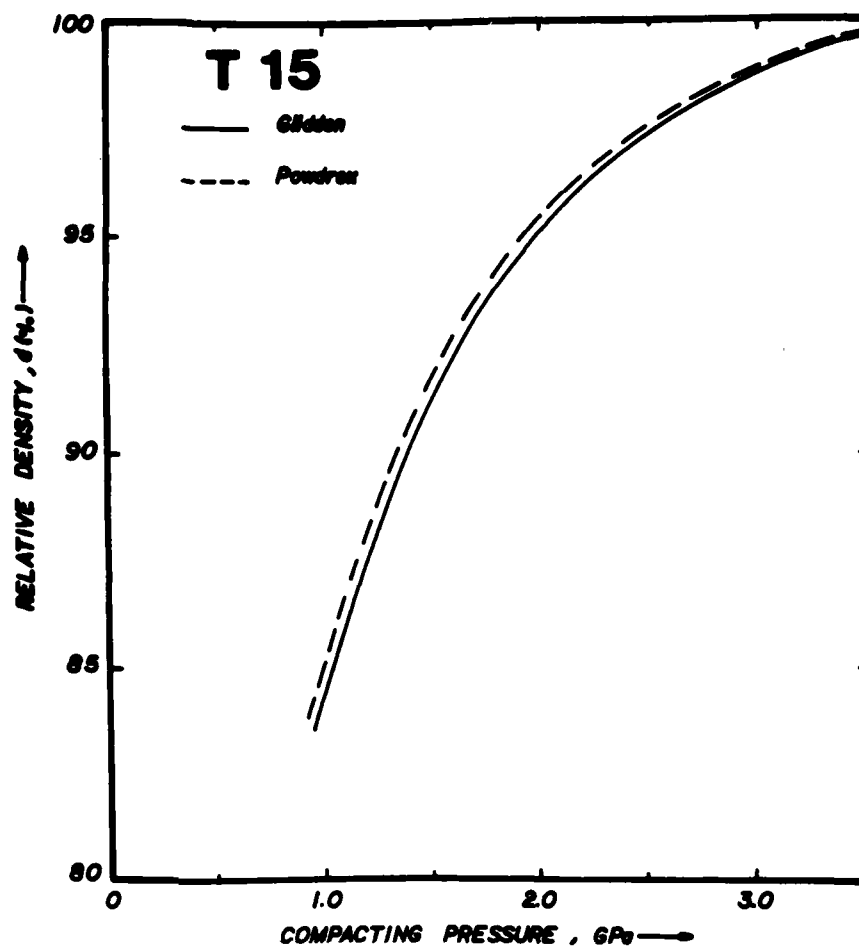
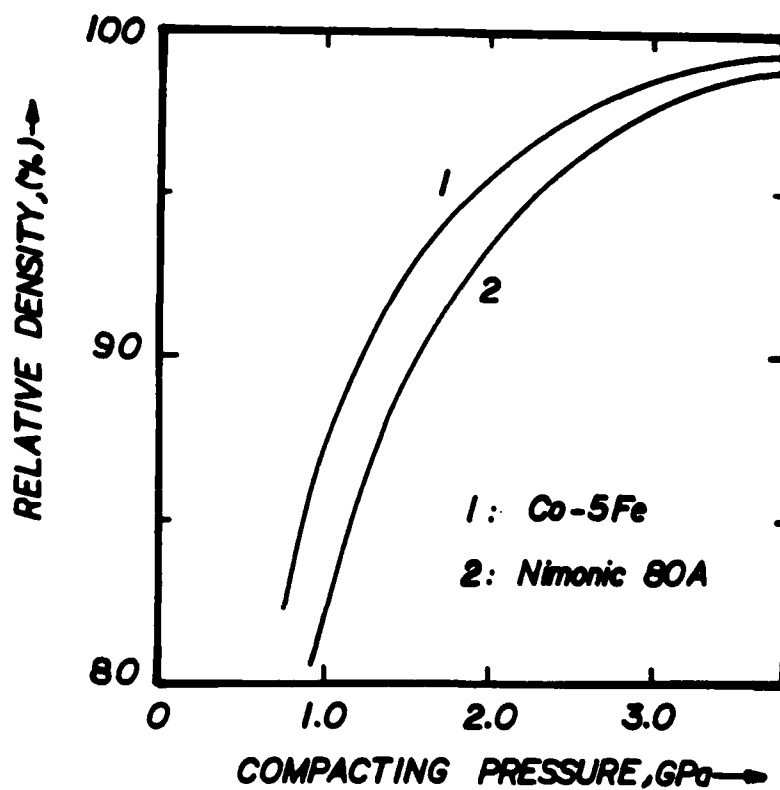


Fig. 4. Compaction curves for water atomized Co-5Fe alloy and Nimonic 80A powders.



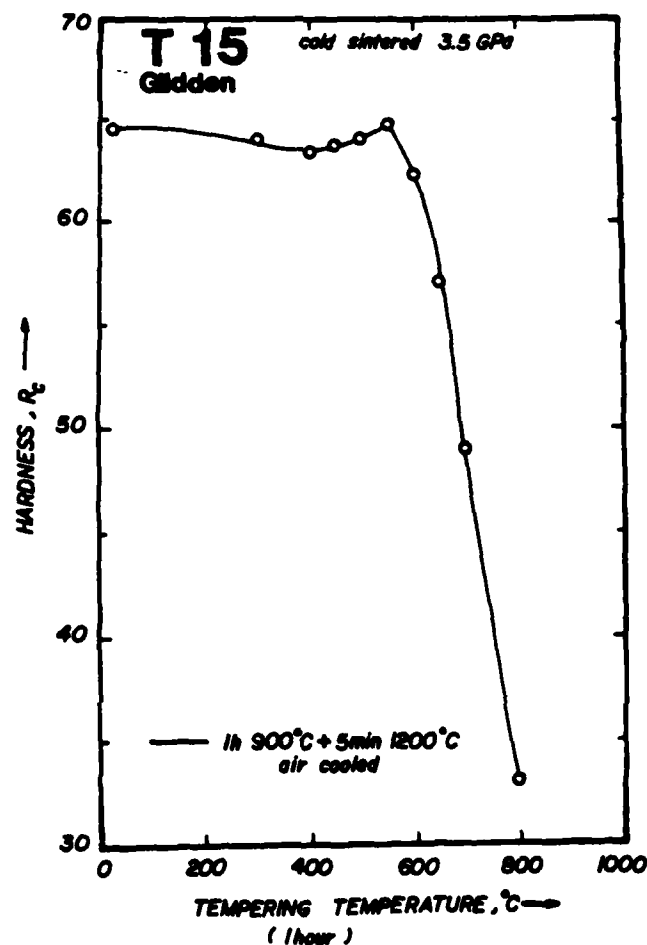


Fig. 5. Tempering curve for P/M T15 (Glidden) high speed tool steel, cold sintered at $P = 3.5$ GPa, annealed 1 hour at 900°C, heat treated for 5 min. at 1200°C and air-cooled.

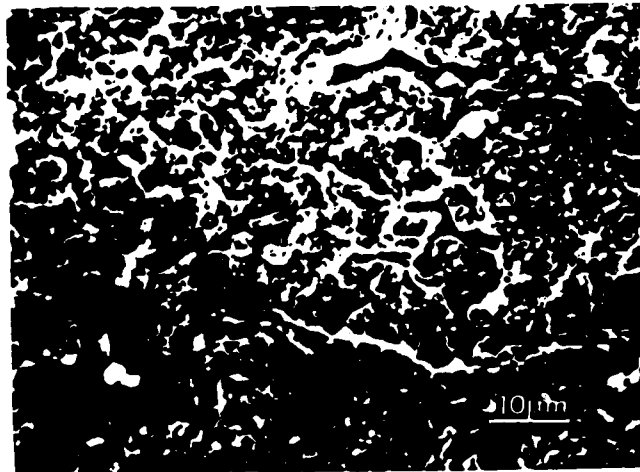


Fig. 6. Fracture surface (SEM) of cold sintered P/M Ti5. $P = 3.5$ GPa; annealed 1 hour at 900°C , austenitized 5 min. at 1200°C , air-cooled and tempered 2×1 hour at 550°C .

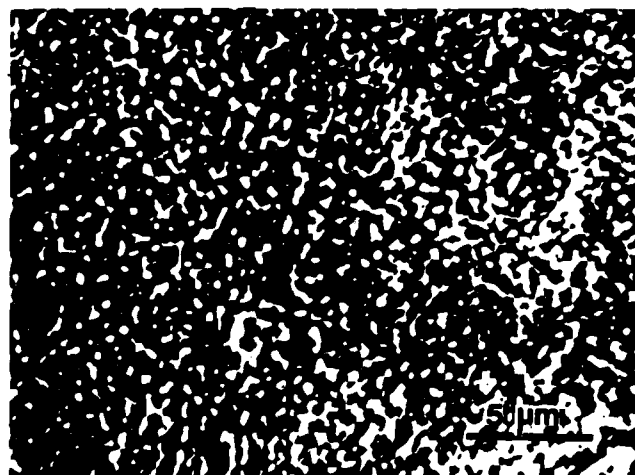


Fig. 7. Microstructure (SEM) of cold sintered P/M Ti5. Cold sintered, $P=3.5$ GPa. Kalling's Etch.

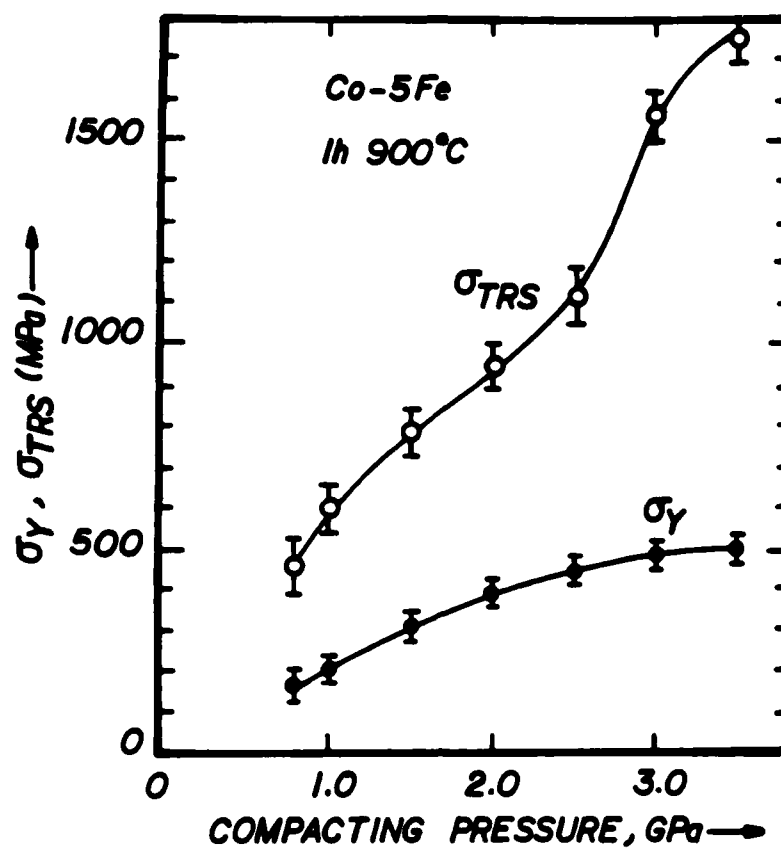


Fig. 8. Dependence of yield stress (σ_y) and transverse rupture strength (σ_{TRS}) on compacting pressure for Co-5Fe water atomized powder.

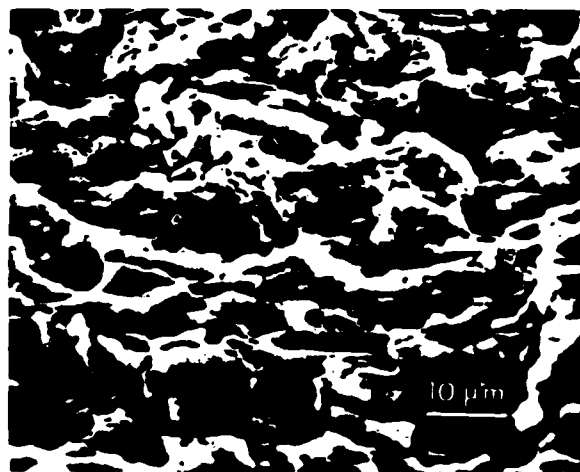


Fig. 9. Fracture surface (SEM) of Co-5Fe alloy cold sintered at $P = 3.0$ GPa and annealed for 1 hour at 900°C.

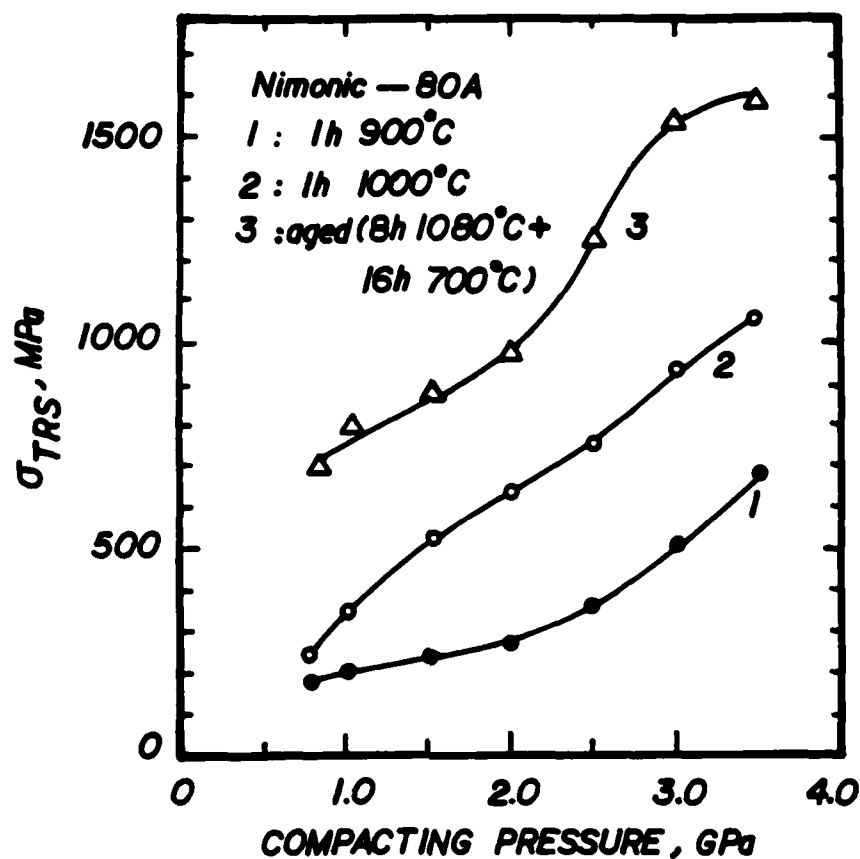


Fig. 10. Dependence of transverse rupture strength (σ_{TRS}) on compacting pressure for P/M Nimonic 80A; 1 - annealed 1 hour at 900°C, 2 - annealed 1 hour at 1000°C, 3 - aged 8 hrs. at 1080°C and 16 hrs. at 700°C.

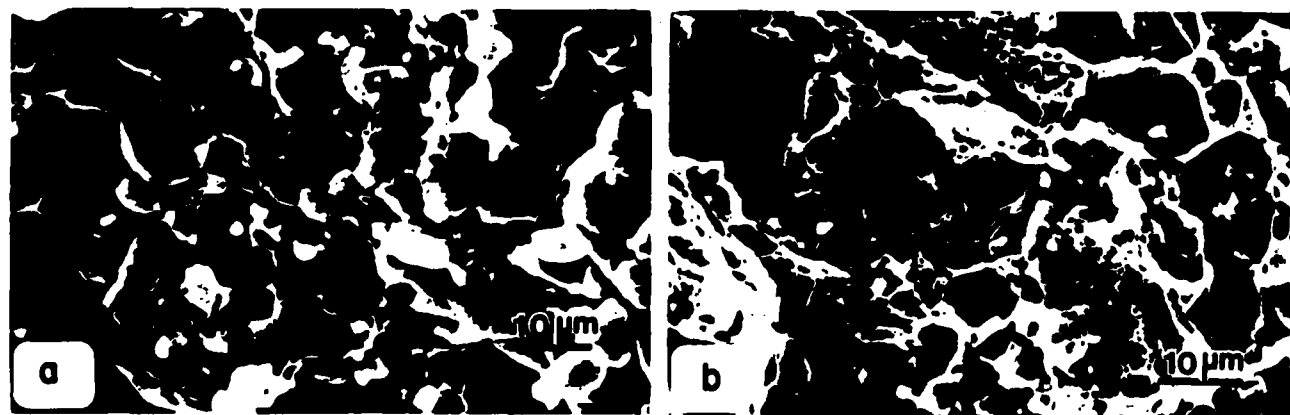


Fig. 11. Fracture surfaces (SEM) of Nimonic 80A powder cold sintered at $P=3.0\text{GPa}$. a) annealed 1 hour at 900°C, b) aged 8 hrs. at 1080°C and 16 hrs. at 700°C.

1. RAPIDLY SOLIDIFIED POWDERS (METASTABLE STRUCTURES)

COLD SINTERING (OR WARM SINTERING)

(HIGH PRESSURE CONSOLIDATION)



HEAT TREATMENT

(ANNEALING,QUENCHING OR AGING)



PART- FINAL SHAPE AND SIZE

2. COMPOSITE MATERIALS (METASTABLE COMPOSITIONS)

ALLOY DESIGN

COLD SINTERING



DIFFUSION ALLOYING

(RELATIVELY LOW TEMPERATURES)



HEAT TREATMENT



PART- FINAL SHAPE AND SIZE

3. HIGH PERFORMANCE PARTS,MASS PRODUCTION

COMPACTION



ANNEALING IN REDUCING ATMOSPHERE



COLD SINTERING



DIFFUSION ALLOYING



REPRESSING (OR HIP)



HEAT TREATMENT



PART- FINAL SHAPE AND SIZE

Fig. 12. Possible processing routes using the cold sintering technique.

END

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